PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

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Serial No.: 09/748,529

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For: UV- OR HEAT-TRIGGERED LOW OXYGEN PACKAGING SYSTEM EMPLOYING AN OXIDIZABLE POLYMER RESIN AND A PEROXIDE

Confirmation No.: 4199

Group Art Unit: 1714

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Attorney Docket: 2039.006200/RFE

CUSTOMER NO. 37774

SECOND DECLARATION UNDER 37 C.F.R. § 1.131 OF ANNE EBBESEN

I, ANNE EBBESEN, declare as follows:

1. At a time prior to November 3, 2000, I was a coworker of Gunnar Rysstad, who is named as an inventor of the present patent application, identified above. At that time, we were employed at Elopak a.s., a corporation organized under the laws of Norway, and having a street address at Grevegården 24, 1369 Stabekk, Norway.

2. Exhibit 1 attached hereto, which includes 6 pages, is a true copy of a project report written by me after January 1, 1996 and before November 3, 2000. The actual date has been blacked out.

3. In Exhibit 1, I report that I tested the initiation of oxygen scavenging in a carton, the structure of which contained an oxygen scavenging polymer ("OSP") sample provided by Chevron Chemical Company ("Chevron"). This test was performed at Mr.

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Rysstad's request. One example of the testing process involved the following steps, performed at an Elopak facility in Norway after January 1, 1996 and before November 3, 2000: spraying a 2% H_2O_2 solution into the interior of the carton; exposing the carton interior to UV light (at a dosage from 306 mJ/cm^2 to 2931 mJ/cm^2); and filling the carton with water (Exhibit 1, pp. 2-3, sections 2.2-2.3, Figure 1, Table 2). The Test also comprised, after exposing the film to UV light, drying the carton interior with hot air. Headspace oxygen and dissolved oxygen in the carton were measured as described in Exhibit 1, p. 4, section 2.4

4. The results of the Test are shown in Exhibit 1, pp. 4-5, sections 3.1-3,2, Figures 2-3. The carton's consumption of both headspace oxygen and dissolved oxygen proceeded more rapidly the longer the duration (and correspondingly, the greater the dosage) of UV exposure to the cartons. (In the figure captions, the durations are given after the slash, as "x Seconds." The "UV On" and "UV Off" statements before the slash indicate whether additional UV exposure took place during conversion of the carton raw material into the carton but prior to spraying of the H_2O_2 solution into the carton interior; see Exhibit 1, p. 2, section 2.1).

5. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

AEB

Date: 06/02/04

Anne Ebbesen

ANNE EBBESEN

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RESEARCH AND DEVELOPMENT DEPARTMENT

PROJECT REPORT

SECURITY CLASS:	**Confidential**
REPORT NO:	**1679**

TITLE:	Verification of film activity on alu-carton with oxygen scavenging polymer(OSP)
DATE OF REPORT:	
PROJECT NO:	P0030
AUTHOR(S):	Anne Ebbesen
RESOURCE CENTRE NAME:	Food Science & Technology Department
RESOURCE CENTRE NO:	702510
KEYWORDS:	Alu, oxygen,chemical analysis

ABSTRACT:

The experiments carried out show that the alu-cartons with oxygen scavenging polymer(OSP) is active in removing oxygen, both from water and from air. At least 30 seconds exposure time to UV light is needed to activate the OSP system. The lag time before the reaction speeds up is decreasing when increasing exposure time to UV light.

DISTRIBUTION: Gunnar Rysstad, Keith Johnstone,Jørn Olsen

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Verification of film activity on alu-carton with oxygen scavenging polymer(OSP)

1. objective

The objective of the work was to:
1.Evaluate which UV dosages was needed to activate the OSP system
2. Determine the activity of the oxygen scavenging system

2. materials and methods

2.1 background information

The coated reel was divided into two different part, one that had been coated with corona-treatment of the Al foil and one part without treatment. It was noted by Chevron that the adhesion might be insufficient on the part without corona treatment.
The reel was converted at converter E (ATN) in Speyer. On the converter there are two stations for using UV-light(Wave length 200-280 nm) on the outside of the board. Output power of the lamp is up to 200W/ cm^2 , and the dose on the outside(print side) of the carton is 140 mJ/ cm^2. The last one was turned off for both part off the reel, and the first was on during the first part and off on the second part. Since OSP system is activated by UV light we had to check if UV light on during converting had any effect on the activity of the OSP system.

2.2 activation of cartons

The alu-cartons with OSP were activated by running the cartons through the aseptic rig. The aseptic rig is shown in Figure 1. The cartons were sprayed with 2% H_2O_2, exposed to UV light for different exposure times and dried with hot air. Table 1 show which combination of parameters that were tested.The settings for hydrogen peroxide flow and hot air temperature was as on an aseptic filling machine U-S80A. The UV Source was a Heraeus NG7087 lamp with λ_{max} 253.7. The dosage of the UV light was measured at a distance of 57 mm from the light source to the sensor of the Solascope UV Intensity meter. Parameters for UV activation is shown in Table 2.

Table 1 An overview of different combination of parameters that were tested

Exposure time to UV light / UV off/on during converting	6 seconds	13 seconds	30 seconds	60 seconds
ON	✔	✔	✔	
OFF	✔	✔	✔	✔

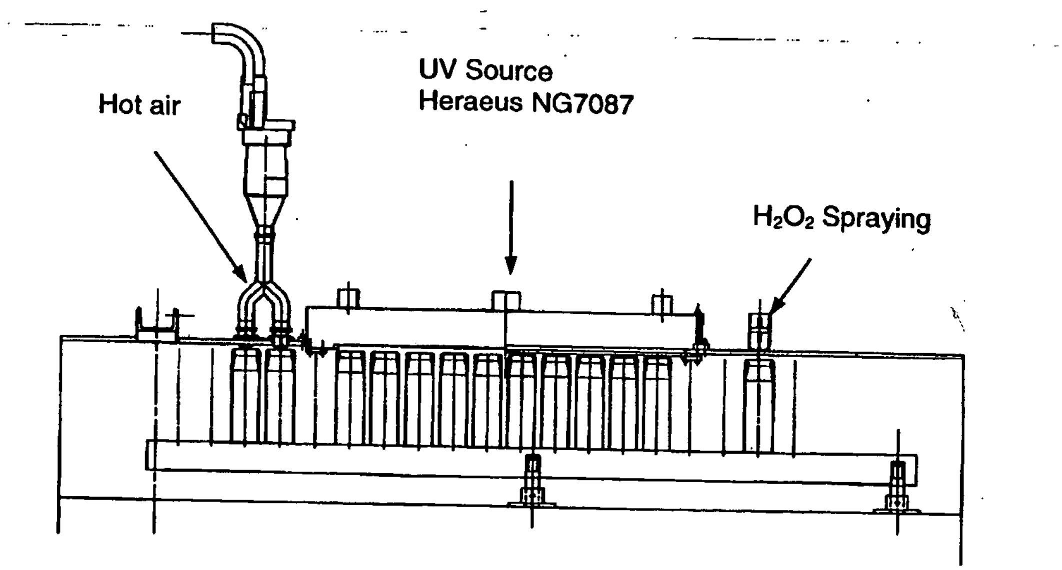

Figure 1 Set up for UV activation

Table 2 Parameters for UV activation of alu-cartons with OSP

	Exposure time 6 seconds	Exposure time 12 seconds	Exposure time 30 seconds	Exposure time 60 seconds
Dosage (mJ/cm^2)	306	612	1510	2931

2.3 experimental design

After activation, the cartons was filled with cold water on a Pure Pak P-S30 filling machine at the R & D system application centre, in Lier, Norway. The water was filled into 1/1 L alu-cartons with OSP with the structure 14 PE / 272 CTMP / 18 PE / 21 Al / Tie / OSP / PE2. The cartons were stored at roomtemperature(RT). All chemical analysis were performed in triplicates i.e. from minimum three separate cartons for each variable.

2.4 methods for analysis of removal of oxygen

Headspace oxygen

A septum was glued onto the surface of the carton and 0.3 ml gas samples were drawn out by using a gastight syringe (Pressure Lok Series A-2 Gassyringes, Dynatech). The stopvalve was closed and the gas compressed to 0.05 ml. The stop valve is opened and the gas sample is injected into a gas chromatograph, Hewlett Packard HP 5890A to separate O_2 and N_2. The column used was a 30 m x 0.32 mm i.d HP PLOT Molecular Sieve 5A with 12 µm film thickness. The oven temperature was 60 °C and oxygen was detected by a thermal conductivity detector. Argon (99.999%) was used as carrier gas.

Dissolved oxygen

Dissolved oxygen in water was measured by using a Micro O_2 Logger from Orbisphere Laboratories.

3. results

3.1 activation of osp system

The removal of dissolved oxygen from water and headspace oxygen is shown in Figure 2 and Figure 3. These figures show that at least 30 seconds exposure to UV light is needed for activation of the OSP system, but even then the rate of removing oxygen is slow . After exposure to UV light for 30 seconds the system has a lag time before it starts to remove oxygen from headspace and from water. The lag time of the OSP system decreases when increasing the exposure time to UV light.

3.2 removal of oxygen

The changes in dissolved oxygen content in water and oxygen in headspace during the test period is shown in Figure 2 and Figure 3. Oxygen is removed from water and headspace at different rates depending on the exposure time to UV light. The lowest exposure times 6 and 13 seconds are not enough to activate the OSP system, and therefore no oxygen is removed neither from water nor from headspace. Increasing exposure time to 30 seconds is activating the OSP system and the system starts to remove oxygen from water and headspace after 3-6 days. The cartons with UV on during converting are removing oxygen with a slower rate than the cartons that had UV off during converting. After 16 days the level of dissolved oxygen and headspace oxygen in cartons with UV off and in cartons with UV on during converting is nearly the same, about 3-4 mg/l for dissolved oxygen in water and 9-10% oxygen in headspace. The lag time of the OSP system decreases when increasing the exposure time to UV light to 60 seconds. The system starts to remove headspace oxygen at day one. Increasing exposure time to 60 seconds is also increasing the rate of removing oxygen, both from water and from headspace. The dissolved oxygen content decreased from 7.5 to 1 7 mg/l in 10 days and

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headspace oxygen decreased from 20.9 to 5.4% in 10 days.

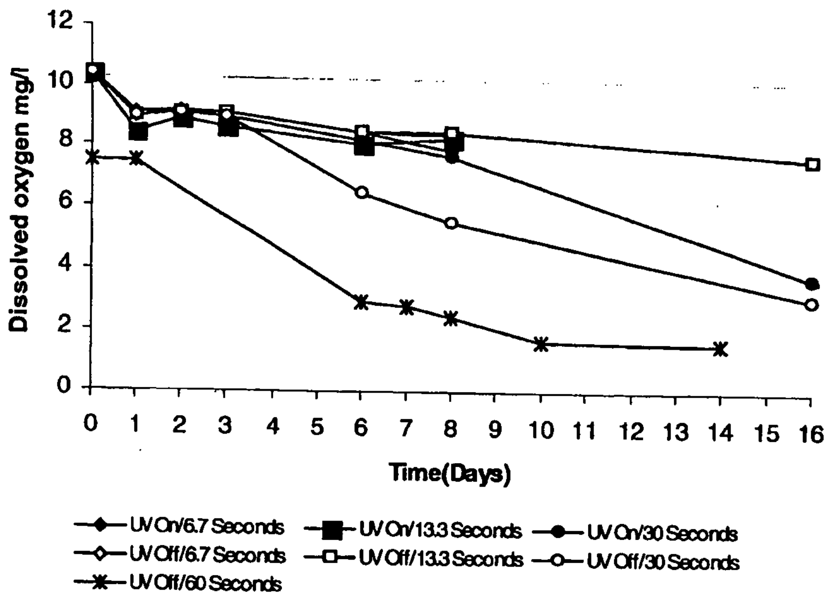

Figure 2 Changes in dissolved oxygen in water during test period

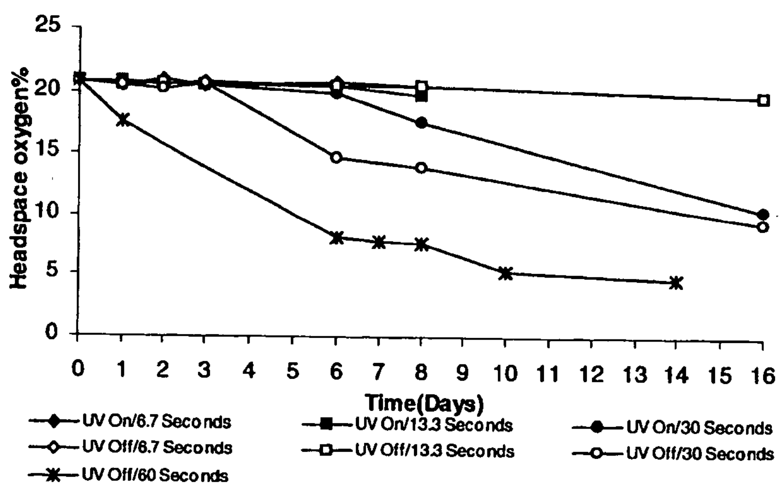

Figure 3 Oxygen measured in headspace during test period

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4. conclusion

The experiments carried out show that the alu-cartons with oxygen scavenging polymer(OSP) is active in removing oxygen, both from water and from air. At least 30 seconds exposure time to UV light is needed to activate the OSP system. The lag time before the reaction speeds up is decreasing when increasing exposure time to UV light.